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IS 8626 (1987): R Salt, Technical [PCD 9: Organic Chemicals Alcohols and Allied Products and Dye Intermediates]



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Bhartrhari—Nitiśatakam

“Knowledge is such a treasure which cannot be stolen”

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IS : 8626 - 1987

Indian Standard
SPECIFICATION FOR
R SALT, TECHNICAL
(*First Revision*)

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BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

Indian Standard

SPECIFICATION FOR R SALT, TECHNICAL

(First Revision)

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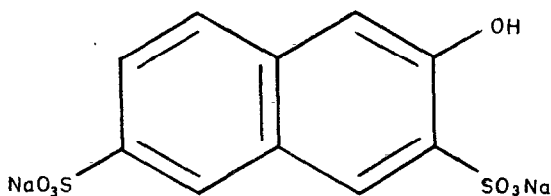
Indian Standard

SPECIFICATION FOR R SALT, TECHNICAL (First Revision)

0. FOREWORD

0.1 This Indian Standard (First Revision) was adopted by the Indian Standards Institution on 31 March 1987, after the draft finalized by the Dye Intermediates Sectional Committee had been approved by the Petroleum, Coal and Related Products Division Council.

0.2 R Salt ($C_{10}H_6O_7S_2Na_2$) is a dye intermediate. It is disodium salt of 2-naphthol-3, 6-disulphonic acid. It is represented by the following structural formula:



R SALT

(Molecular Mass, Free Acid, 304)

0.3 This standard was first published in 1977. It has now been revised in order to update it. In the present version the requirement of assay (on the basis of molecular mass 304), β -naphthol and matter insoluble in sodium hydroxide have been modified. Besides, the requirement of impurities, namely, R Salt and Schaeffer's salt have been stipulated and chromatographic test method for their estimation has been introduced.

0.4 For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS : 2-1960*. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

*Rules for rounding off numerical values (revised).

1. SCOPE

1.1 This standard prescribes the requirements and methods of sampling and test for R salt, technical.

2. REQUIREMENTS

2.1 Description — The material shall be in the form of off-white powder or off white moist cake.

2.2 The material shall also comply with the requirements given in Table 1.

TABLE 1 REQUIREMENTS FOR R SALT, TECHNICAL

SL No.	CHARACTERISTIC	REQUIREMENT	METHOD OF TEST, REF TO CL No. IN APPENDIX A
(1)	(2)	(3)	(4)
i)	Assay, percent by mass (on dry basis), <i>Min</i>	65	A-2
ii)	Matter insoluble in sodium hydroxide solution, percent by mass, <i>Max</i>	0.2	A-3
iii)	β -Naphthol content, percent by mass (on 100 percent basis), <i>Max</i>	0.5	A-4
iv)	G salt, percent by mass (on 100 percent basis), <i>Max</i>	1.0	
v)	Schaeffer's salt, percent by mass (on 100 percent basis), <i>Max</i>	1.0	

3. PACKING AND MARKING

3.1 Packing — The material shall be packed in steel drums (see IS : 2552-1979*) lined with suitable polyethylene film or as agreed to between the purchaser and the supplier. Each container shall be securely closed.

*Specification for steel drums (galvanized and ungalvanized) (second revision).

3.2 Marking — Each container shall bear legibly and indelibly the following information:

- a) Name of the material;
- b) Name of the manufacturer and his recognized trade-mark, if any;
- c) Batch number; and
- d) Net, gross and tare mass.

3.2.1 The containers may also be marked with the Standard Mark.

NOTE — The use of the Standard Mark is governed by the provisions of the Bureau of Indian Standards Act, 1986 and the Rules and Regulations made thereunder. The Standard Mark on products covered by an Indian Standard conveys the assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control which is devised and supervised by BIS and operated by the producer. Standard marked products are also continuously checked by BIS for conformity to that standard as a further safeguard. Details of conditions under which a licence for the use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

4. SAMPLING

4.1 Representative samples of the material shall be drawn as prescribed in 3 of IS : 5299-1969*.

4.2 Number of Tests

4.2.1 Test for assay shall be conducted on each of the individual sample.

4.2.2 Tests for the determination of the remaining characteristics, namely, β -naphthol content, solubility in sodium hydroxide solution, G salt and Schaeffer's salt content shall be conducted on the composite sample.

4.3 Criteria for Conformity

4.3.1 *For Individual Samples* — The lot shall be declared as conforming to the requirement of assay if each of the individual test results satisfies the relevant requirement given in Table 1.

4.3.2 *For Composite Sample* — For declaring the conformity of the lot to the requirements of all other characteristics tested on the composite sample (see 4.2.2), the test results for each of the characteristics shall satisfy the relevant requirement given in Table 1.

*Methods of sampling and tests for dye intermediates.

5. TEST METHODS

5.1 Tests shall be carried out according to the methods prescribed in Appendix A. Reference to relevant clauses of Appendix A is given in col 4 of Table 1.

5.2 Quality of Reagents — Unless specified otherwise, pure chemicals and distilled water (*see* IS : 1070-1977*) shall be employed in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A P P E N D I X A

(Table 1 and Clause 5.1)

METHODS OF TEST FOR R SALT, TECHNICAL

A-1. PREPARED SAMPLE

A-1.1 Dry the material at $105 \pm 1^{\circ}\text{C}$ to constant mass. Grind and mix well. Transfer the material to a wide-mouthed bottle and stopper it. Do not expose the sample to an atmosphere containing acidic or alkaline fumes. Use this *prepared sample* for tests.

A-2. ASSAY

A-2.0 Outline of the Method — The material is dissolved in water adding sodium carbonate solution. A known volume of the solution is titrated against standard 4-chlorobenzene diazo solution in alkaline medium and from the consumption of the diazonium solution, assay is calculated.

A-2.1 Reagents

A-2.1.1 Sodium Carbonate Solution — 10 percent (*m/v*).

A-2.1.2 Phenolphthalein Indicator Paper

A-2.1.3 Standard 4-Chlorobenzene Diazo Solution — 0.1 N.

A-2.1.4 H Acid Indicator — Dissolve 0.5 g of H acid in 100 ml of 1 percent ammonium hydroxide solution.

A-2.2 Procedure — Weigh accurately about 10 g of the *prepared sample* (*see* A-1.1) and transfer to a 500-ml beaker with the help of water. Slowly add sodium carbonate solution to get a positive test on

*Specification for water for general laboratory use (*second revision*).

phenolphthalein paper. Transfer the solution quantitatively into a 500-ml volumetric flask and dilute to the mark with water at room temperature. Mix well. Pipette 100 ml aliquot of this solution into a 1-litre beaker. Add 200 ml of ice-cold water. Add 50 ml of sodium carbonate solution. Stir mechanically and cool with washed ice to below 1°C. Titrate with 4-chlorobenzene diazonium solution from a cold water-jacketted burette. Test with H acid for the excess of diazo and with the diazo for the coupling component. Add 50 g of pure sodium chloride towards the end point. The end point is noted when there is no test with diazo and a fine visible pink line with H acid indicator is obtained which may persist for 10 minutes without further addition of diazo. Let the titre reading be V .

A-2.3 Calculation

$$\text{Assay, percent by mass} = \frac{V \times N \times 152}{M}$$

where

V = volume in ml of the standard diazonium solution,

N = normality of 4-chlorobenzene diazo solution, and

M = mass (on dry basis) in g of the material taken for the test.

A-3. DETERMINATION OF MATTER INSOLUBLE IN SODIUM HYDROXIDE SOLUTION

A-3.1 Reagent

A-3.1.1 Sodium Hydroxide Solution — Approximately 5 percent (m/v) filtered free from suspended impurities.

A-3.2 Procedure — Weigh accurately 10 to 15 g thoroughly mixed sample into a 1 000-ml beaker, add 300 ml water and sufficient 5 percent sodium hydroxide solution to make the solution alkaline to brilliant yellow paper. Heat the solution to 60 C until the sample is dissolved and filter it through sintered crucible of porosity G4, wash residue well with hot water, dry at $100 \pm 5^\circ\text{C}$, cool and weigh.

A-3.3 Calculation

$$\begin{array}{l} \text{Matter insoluble in sodium hydroxide} \\ \text{solution, percent by mass} \end{array} = \frac{M_1 \times 100}{M_2}$$

where

M_1 = mass in g of the residue, and

M_2 = mass in g of sample taken for the test.

A-4. DETERMINATION OF G SALT AND SCHAEFFER'S SALT AND β -NAPHTHOL

A-4.0 Outline of the Method — G salt, Schaeffer's salt and β -naphthol are determined by using descending paper chromatographic technique.

A-4.1 Apparatus

A-4.1.1 *Developing Chamber*

A-4.1.2 *Chromatographic Sprayer*

A-4.1.3 *Micropipette*

A-4.1.4 *UV Lamp*

A-4.2 Reagents

A-4.2.1 *G Salt* — 0.1 percent solution (on 100 percent basis) in water + 3N ammonia solution (9 : 1).

A-4.2.2 *Schaeffer's Salt* — 0.1 percent solution (on 100 percent basis) in water + 3N ammonia solution (9 : 1).

A-4.2.3 *β -Naphthol* — 0.05 percent solution (on 100 percent basis) in water + 3N ammonia solution (9 : 1).

A-4.2.4 *Developer* — 40 percent aqueous solution of calcium chloride.

A-4.2.5 *R Salt* — Free from G salt, Schaeffer's salt and β -naphthol.

A-4.2.6 *Spray Reagent* — 0.1 percent aqueous solution of diazo fast red B-Salt.

A-4.2.7 *Sodium Hydroxide Solution* — 2 percent.

A-4.3 Procedure

A-4.3.1 First, prepare a standard solution of R salt containing known amount of G salt, Schaeffer's salt and β -naphthol. Into each of these 100-ml volumetric flasks, weigh accurately 1.0 g of R salt (**A-4.2.5**). Add 8.0 ml, 9.0 ml and 10.0 ml of 0.1 percent solution of G salt (**A-4.2.1**) in ammonia solution to flask No. 1, 2 and 3 respectively. Dissolve the contents of the flasks in ammonium hydroxide solution. Make up the volume to the mark with ammonium hydroxide solution. Now, there are 3 solutions of 0.8, 0.9 and 1.0 percent G salt content. In a fourth 100-ml flask, weigh accurately about 1.0 g of the *prepared sample* (see **A-1.1**), dissolve in ammonium hydroxide solution and dilute 100 ml with ammonium hydroxide solution.

A-4.3.1.1 Prepare a similar set of 3 solutions of 0.8, 0.9 and 1.0 percent Schaeffer's salt content with the use 0.1 percent Schaeffer's salt solution in ammonia (**A-4.2.2**).

A-4.3.1.2 Prepare a similar set of 3 solutions of 0.4, 0.45 and 0.5 percent of β -naphthol content with the use of 0.05 percent β -naphthol solution in ammonia (**A-4.2.3**).

A-4.3.2 Place 10 microlitre spot of each of the three solutions of G salt, Schaeffer's salt and β -naphthol and *prepared sample* solution (**A-4.3.1**) using micropipette in the same line to a distance of about 4 cm on filter paper (Whatman No 1 or equivalent). Place the paper in a descending paper chromatographic glass jar containing the developing reagent (**A-4.2.4**) and previously saturated with the same developer. Allow the solvent to run in a descending manner for about 30 cm from the spot. This will take about 7 hours. Take out the paper after 30 cm run, dry the solvent completely and observe under UV light. Spray the paper with spray reagent (**A-4.2.6**) and develop the chromatogram.

The separated spots characteristic of constituents may be identified by their colour as under:

Contents	Zone	Rf Value	Colour	
			Under UV Light	After Spraying
β -Naphthol	I	0.16	—	Pink
Schaeffer's Salt	II	0.33	—	Pink
G Salt	III	0.62	Blue	Pink
R Salt	IV	0.72	Blue	Pink
Unknown	V	0.89	Green	—

A-4.4 Report — Report G salt, Schaeffer's salt and β -naphthol as that which is nearest in intensity to the standard. In case the colour intensity does not come in the range of standard spots, repeat the whole procedure using different percentage of G salt, Schaeffer's salt and β -naphthol.

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	$1 \text{ N} = 1 \text{ kg.m/s}^2$
Energy	joule	J	$1 \text{ J} = 1 \text{ N.m}$
Power	watt	W	$1 \text{ W} = 1 \text{ J/s}$
Flux	weber	Wb	$1 \text{ Wb} = 1 \text{ V.s}$
Flux density	tesla	T	$1 \text{ T} = 1 \text{ Wb/m}^2$
Frequency	hertz	Hz	$1 \text{ Hz} = 1 \text{ c/s (s}^{-1}\text{)}$
Electric conductance	siemens	S	$1 \text{ S} = 1 \text{ A/V}$
Electromotive force	volt	V	$1 \text{ V} = 1 \text{ W/A}$
Pressure, stress	pascal	Pa	$1 \text{ Pa} = 1 \text{ N/m}^2$